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Mines Branch Investigation Report IR 74-44

BENEFICIATION OF GRAPHITE FROM MONT LAURIER, QUEBEC

(PROJECT MP-IM-7106)

by

F. H. Hartman* and R. A. Wyman**

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SUMMARY

Beneficiation trials were made on a graphite sample from Mont Laurier, Quebec containing 17.4% carbon, which included self-grinding, air tabling, flotation, and magnetic separation.

Air tabling produced a graphite concentrate of 78.4% carbon, representing a recovery of 12% of the carbon in the feed.

Flotation with (1) minus 8-mesh feed from self-grinding, followed by screening to separate the product, gave a plus 65-mesh concentrate containing 95.4% carbon for a recovery of 30% of the original carbon, and (2) the same feed, ground in a ball mill, gave a plus 65-mesh product containing 93.5% carbon, for a recovery of 46%; the removal of a magnetic fraction was used to upgrade the concentrates.

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Direction des Mines

Rapport d'Investigation IR 74-44

ENRICHISSEMENT DE GRAPHITE PROVENANT DE MONT-LAURIER, QUEBEC

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RESUME

Les auteurs ont procédé à des essais de concentration par broyage autogène, concentration par tables pneumatiques, flottation et séparation magnétique sur un échantillon de graphite de 17.4% de carbone provenant de Mont-Laurier, Québec.

On obtint, par concentration par tables pneumatiques, un concentré de graphite de 78.4% de carbone, ce qui représente une récupération de 12% de carbone basé sur l'alimentation.

Ensuite, (1) par le processus de flotation avec une alimentation moindre que 8 mailles obtenue par broyage autogène, suivie d'un tamisage du produit, on obtint un concentré supérieur à 65 mailles contenant 95.4% de carbone totalisant une récupération de 30% du carbone original. (2) Avec la même alimentation, mais cette fois fragmentée dans un broyeur à boulets, on obtint un produit supérieur à 65 mailles contenant 93.5% de carbone, d'une récupération de 46%; le rejet d'une fraction magnétique fut utilisé afin d'améliorer la teneur des concentrés.

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INTRODUCTION

A request was received August, 1971, from MHM Syndicate, Ottawa, for an investigation of material from a graphite deposit near Mont Laurier, Quebec. In September, J. E. Hayes, MHM Syndicate, accompanied by Frederick H. Murphy, The Asbury Graphite Mills, Inc., Asbury, N.J., visited the Mines Branch to discuss the problem. Murphy expressed interest in the product; no "flake" graphite was available in North America at that time.

In 1958, detailed beneficiation tests were made on samples of weathered graphite from the Mont Laurier area[1]. As those samples may not have been from the same deposit, it was decided that in the present study a preliminary investigation would be done with new material; a 300 pound sample was sent to the Mines Branch in September of 1971 for testing. Thin sections of representative material revealed that the sample was composed chiefly of calcite and quartz, about 15% of graphite, and small amounts of mica and iron oxides.

PROCEDURES

Analysis

The graphite content of samples was determined by acid leaching of solubles followed by loss on ignition (LOI) analysis of the residue. Various times and temperatures of ignition were tried; the most accurate procedure found was to heat the samples for 4 hours at 900°C in a muffle furnace with positive air circulation. Re-runs were made to check the early analyses obtained under somewhat different conditions.

The products were examined under the microscope to detect free graphite. However, it was almost impossible to differentiate between graphite and free biotite.

Preparation of Feed for Tests

The nature of this sample suggested that it would be amenable to self-grinding, i.e., using larger lumps of feed to grind smaller sizes. To test the possibility, approximately one third of the sample received was separated into plus 3-in. lumps and minus 1-in. fines. A small, 60-rpm, laboratory ball mill, 11 in. (28 cm) in diameter and 7 in. (18 cm) in length, was used for grinding.

For the initial load, 10 pounds of plus 3-in. lump and 10 pounds of minus 1-in. fines were used. The minus 8-mesh fraction was removed prior to grinding, to prevent over-grinding of coarse graphite flakes. The mill was operated for 15 minutes, then was dumped and the contents separated into plus 1-in., minus 1-in., and minus 8-mesh fractions. For subsequent cycles the charge was supplemented as required, then ground, dumped and sized; this method was followed through 10 complete cycles. The results are compiled in Table 1.

The minus 8-mesh product from self-grinding was used as feed throughout the investigation.

Magnetic Separation

To check the possibility of concentration of graphite by removing a magnetic product, the minus 8-mesh feed from grinding was screened on a 28-mesh sieve and the fine fraction was passed through a Jones wet magnetic separator at a setting of 25 amperes. The plus 28-mesh fraction, too coarse for the Jones equipped with high-intensity plates, was analysed without magnetic treatment. Results of the separation are given in Table 2 together with carbon and acid soluble analyses.

TABLE 1

Details of Ten Cycle Self-grinding Test

Cycle No.	Grinding Time (min)	Charge to Mill (lb)				Total Charge	-8 mesh Product (lb)	Totals (lb)
		+1 in.		-1 in.+8 mesh				
		Old	New	Old	New			
1	15	8.50	10.00	3.25	8.50	18.50	1.50 6.75	20.00 18.50
2	15	8.50 7.75		3.25 4.25	6.50	18.25	1.75 6.25	20.00 18.25
3	15	7.75 7.75	1.25	4.25 5.25	5.50	18.75	1.25 5.75	20.00 18.75
4	20	7.75 8.50	1.00	5.25 3.75	5.25	19.25	0.75 7.00	20.00 19.25
5	20	8.50 6.50		3.75 5.75	5.50	17.75	2.25 5.50	20.00 17.75
6	20	6.50 7.75	2.25	5.75 5.25	4.75	19.25	0.75 6.25	20.00 19.25
7	20	7.75 7.25	1.00	5.25 6.00	5.00	19.00	1.00 5.75	20.00 19.00
8	20	7.25 7.25	1.50	6.00 6.50	4.25	19.00	1.00 5.25	20.00 19.00
9	20	7.25 8.25	1.75	6.50 5.25	3.50	19.00	1.00 5.50	20.00 19.00
10 *	20	8.25 8.50	0.75	5.25 5.00	4.75	19.00	1.00 5.50	20.00 19.00
Totals Less*	185		19.50 8.50		53.50 5.00		71.75	
Used			11.00		48.50			
Per Cycle	18.5		1.10		4.85		7.18	

*Remaining after 10 cycles.

TABLE 2

Analyses of Magnetic Fractions from Minus 8-mesh Feed
Jones Test 1, 25 amperes, high-intensity plates

Fraction	% Wt	% Carbon (LOI)		% Acid Soluble	
		Anal	Dist	Anal	Dist
+28 mesh*	32.0	19.32	37.6	29.55	26.1
-28 mesh					
Non-magnetic	21.9	21.75	29.1	38.78	23.5
Middling	29.7	16.13	29.2	37.76	31.0
Magnetic	16.4	4.08	4.1	43.03	19.4
Head (calcd)	100.0	16.40	100.0	36.22	100.0
Head (analysis)		17.40		35.97	

*Screened out before separation

Screening

The self-ground minus 8-mesh material was separated into screened fractions to minus 325-mesh to determine where the carbon and the free flake graphite occurred. The fractions were examined under the microscope and representative portions were analysed; Table 3 summarizes the results.

In a second test, the plus 28-mesh fraction of the feed was comminuted with rolls to minus 28-mesh size and screened with the minus 28-mesh feed. The results of analysis are given in Table 4.

TABLE 3

Analyses of Screen Fractions, Minus 8-mesh Material
Screen Test 1

Fraction Tyler Mesh		% Wt	% Carbon (LOI)		% Acid Soluble		Composition
Minus	Plus		Anal	Dist	Anal	Dist	
8	14	14.7	16.93	16.0	33.44	13.5	Little free graphite. Unbroken lumps with graphite present.
14	28	16.1	21.46	22.2	28.57	12.7	Some free flake. Flakes with gangue attached.
28	35	9.3	23.06	13.8	25.82	6.6	Much more free flake. Gangue mostly clean.
35	48	9.8	16.76	10.6	25.34	6.8	Free flake, free gangue, some middlings.
48	65	9.9	17.86	11.4	26.77	7.3	Free flake, free gangue.
65	100	9.1	12.89	7.5	30.66	7.7	Free flake, free gangue.
100	150	8.0	12.16	6.3	36.38	8.0	Free flake, free gangue, very few middlings.
150	200	3.8	10.29	2.5	40.81	4.3	Free flake, free gangue, few middlings.
200	325	5.7	9.01	3.3	47.54	7.5	Mostly gangue.
325	-	13.6	7.28	6.4	68.40	25.6	Little graphite. Brown, probably from oxidation. Slime problem.
Head (calcd)		100.0	15.53	100.0	36.31	100.0	

Free flake refers to either graphite and/or biotite.

TABLE 4

Analyses of Screen Fractions, Minus 28-mesh Material

Screen Test 3

Fraction Tyler Mesh		% Wt	% Carbon (LOI)		% Acid Soluble		Composition
Minus	Plus		Anal	Dist	Anal	Dist	
-	28	5.1	46.38	15.5	25.06	3.5	-
28	35	14.2	20.02	18.7	26.23	10.2	Some flake particles combined with gangue.
35	48	14.8	18.53	18.1	26.46	10.8	Mostly free flake.
48	65	14.1	15.22	14.1	27.53	10.7	-
65	100	12.4	12.97	10.6	30.36	10.3	-
100	150	8.8	11.65	6.7	34.73	8.4	-
150	200	6.9	10.69	4.8	39.65	7.5	-
200	325	7.0	8.04	3.7	46.01	8.9	-
325	-	16.7	7.12	7.8	64.95	29.7	-
Head (calcd)		100.0	15.22	100.0	36.40	100.0	

Flotation

Tests were run with 500-gram feed samples under the following conditions:

- Material was floated in a 500-gram Denver Sub-A laboratory flotation cell.
- Pulp densities in the primary or rougher float varied, 10-25% solids, depending on the treatment of the feed before flotation.
- Measured amounts of collector and frothing reagents were added to the agitated pulp in the cell.
- After conditioning by mixing, air was admitted and dispersed through the pulp. The resultant air bubbles with attached mineralization were removed as a froth from the surface (termed rougher concentrate).
- The rougher concentrate, and concentrates from subsequent steps, were returned to the cell, mixed with fresh water, and "cleaned" by refloating.
- Material remaining from each flotation step was removed from the cell and referred to, in sequence, as rougher tailings, cleaner 1 tailings, ... Material floated was referred to as "concentrate".

Unground Feed

Attempts were made to float graphite from minus 8-mesh feed with four different collectors or collector combinations: (a) pine oil, (b) pine oil plus kerosene, two taurates (c) Dripon and (d) Igepon T-33. Results when using the taurates were not encouraging; the rougher tailings had high carbon contents.

The results from two tests (12 and 13) using pine oil alone are given in Tables 5, 6, and 6A. In Test 12, the floated rougher concentrate was agitated in an ultrasonic bath for 30 minutes to disperse agglomerated flake material before being returned to the flotation cell for 3 cleaning steps, then magnetically separated at 25 amperes in the Jones separator equipped with high-intensity plates. Test 13 was similar except that the concentrate was cleaned 5 times, after which the non-magnetic concentrate fraction and the 5th cleaner tailings were combined and screened.

Ground Feed

Minus 8-mesh feed, slurried with 50% water, was ground for periods of 10 and 15 minutes in a small 8.75-in. (22.2-cm) diameter x 9.60-in. (24.4-cm) long laboratory Abbé mill run at 80 rpm, with 3000 grams of 1/2-in. (1.25-cm) "burundum cylpebs" grinding media. The product was floated with (a) pine oil, (b) pine oil plus kerosene, (c) pine oil plus isopropyl alcohol, two taurates (d) Dripon and (e) Igepon T-33. The best results were obtained with pine oil alone.

In Test 14 (Table 7), ground 15 minutes, floated with pine oil, cleaned once, the cleaned concentrate was screened. The treatment was similar for Test 15 (Table 8) but the concentrate was treated further in the Jones magnetic separator set at 25 amperes; the non-magnetic fraction was then screened.

TABLE 5

Flotation Test 12, Analyses of Products

Minus 8-mesh Feed, Unground

REAGENT	lb per ton
Pine oil	0.1 x 4 = 0.4
Rougher	0.1
Cleaner 1	0.1
Cleaner 2	
REMARKS	Rougher concentrate dispersed ultrasonically. Final concentrate magnetically fractionated.

ANALYSES / Product	% Wt	% Carbon (LOI) Anal	% Carbon (LOI) Dist	% Acid Soluble Anal	% Acid Soluble Dist
Magnetics, concentrate	0.2	26.98	0.4	52.64	0.2
Non-magnetics, concentrate	1.9	90.74	13.6	4.71	0.2
Cleaner 3 tailings	10.9	73.29	63.4	14.34	3.6
Cleaner 2 tailings	2.0	50.02	7.8	25.77	1.2
Cleaner 1 tailings	6.6	8.08	4.3	40.81	6.1
Rougher tailings	78.4	1.69	10.5	49.68	88.7
Head (calcd)	100.0	12.59	100.0	43.92	100.0

TABLE 6

Flotation Test 13, Analyses of Products

Minus 8-mesh Feed, Unground

REAGENT	lb per ton
Pine oil	0.1 x 4 = 0.4
Rougher	0.1
Cleaner 1	0.1
Cleaner 2	0.1
Cleaner 3	0.1
Cleaner 4	
REMARKS	Rougher concentrate dispersed ultrasonically. Final concentrate magnetically fractionated.

ANALYSES / Product	% Wt	% Carbon (LOI) Anal	% Carbon (LOI) Dist	% Acid Soluble Anal*	% Acid Soluble Dist
Magnetics, concentrate	0.2	52.97	0.9	29.97	0.1
Non-magnetics, concentrate	1.8	91.62	13.6	2.26	0.1
Cleaner 5 tailings	8.3	85.52	58.5	3.48	0.7
Cleaner 4 tailings	0.6	69.49	3.4	17.29	0.3
Cleaner 3 tailings	0.7	68.29	3.9	18.47	0.3
Cleaner 2 tailings	1.1	53.04	4.8	28.20	0.7
Cleaner 1 tailings	7.8	9.06	5.8	41.02	7.6
Rougher tailings	79.5	1.38	9.1	47.79	90.2
Head (calcd)	100.0	12.12	100.0	42.13	100.0

* Some acid soluble values are subject to error.

TABLE 6A

Flotation Test 13, Analyses of Combined Non-magnetic Concentrate and Cleaner 5 Tailings

Screen Test 2

Fraction Tyler Mesh		%	% Carbon (LOI)		% Acid Soluble	
Minus	Plus	Wt	Anal	Dist	Anal*	Dist
-	28	1.6	97.95	1.8	1.53	0.3
28	35	4.8	97.52	5.5	0.88	0.5
35	48	11.0	97.26	12.5	0.81	1.1
48	65	19.8	93.73	21.7	4.05	9.5
65	100	22.6	84.47	22.3	9.13	24.4
100	150	18.6	75.30	16.3	13.97	30.7
150	200	10.8	75.95	9.6	14.46	18.4
200	325	7.1	81.14	6.7	12.51	10.4
325	-	3.7	84.50	3.6	10.85	4.7
Head (calcd)		100.0	85.69	100.0	8.47	100.0

* Some acid soluble values are subject to error.

TABLE 7

Flotation Test 14, Analyses of Products

Minus 8-mesh Feed, Ground 15 minutes

REAGENT	lb per ton
Pine oil	
Rougher	0.1 x 4 = 0.4
Cleaner 1	0.1
REMARKS	Concentrate screened

Product \ ANALYSES		% Wt	% Carbon (LOI) Anal	% Carbon (LOI) Dist	% Acid Soluble Anal	% Acid Soluble Dist
Concentrate	+28 m	1.4	95.17	9.0	3.74	0.1
"	-28+35 m	1.8	94.61	11.1	4.18	0.2
"	-35+48 m	2.3	91.87	14.4	5.23	0.3
"	-48+65 m	2.7	83.49	15.3	7.02	0.5
"	-65+100 m	2.9	67.11	12.8	10.23	0.7
"	-100+150 m	2.5	52.43	8.6	16.31	1.1
"	-150+200 m	1.9	51.73	6.7	21.63	1.1
"	-200+325 m	1.6	53.91	5.9	25.89	1.1
"	-325 m	2.2	59.18	8.6	31.02	1.8
Concentrate (calcd)		19.3	71.56	92.4	13.72	6.9
Cleaner 1 tailings		4.2	9.32	2.6	39.23	4.3
Rougher tailings		76.5	0.97	5.0	44.41	88.8
Head (calcd)		100.0	14.94	100.0	38.26	100.0

TABLE 8

Flotation Test 15, Analyses of Products

Minus 8-mesh Feed, Ground 15 minutes

REAGENT	lb per ton
Pine oil	
Rougher	0.1 x 4 = 0.4
Cleaner 1	0.1
REMARKS	Concentrate magnetically fractionated; non-magnetics screened.

Product \ ANALYSES	% Wt	% Carbon (LOI) Anal	% Carbon (LOI) Dist	% Acid Soluble Anal	% Acid Soluble Dist
Concentrate					
" Magnetic, pass 1	3.3	17.56	3.8	44.48	3.9
" " pass 2	0.7	46.92	2.2	27.65	0.5
" " pass 3	0.5	67.09	2.2	18.57	0.2
" Non-magnetic, + 28m	1.3	95.59	8.0	3.28	0.1
" " -28+ 35m	1.7	94.82	10.5	3.68	0.2
" " -35+ 48m	2.2	93.88	13.5	3.99	0.2
" " -48+ 65m	2.3	90.91	14.1	4.59	0.3
" " -65+100m	2.1	81.99	11.7	5.72	0.3
" " -100+150m	1.6	73.35	8.0	7.80	0.3
" " -150+200m	1.2	73.08	5.9	9.49	0.3
" " -200+325m	1.1	75.02	5.3	11.27	0.3
" " -325m	1.8	68.91	8.1	21.13	1.0
Concentrate (calcd)	19.8	71.13	93.3	14.70	7.6
Cleaner 1 tailings	5.1	6.63	2.2	44.32	6.0
Rougher tailings	75.1	0.90	4.5	43.61	86.4
Head (calcd)	100.0	15.09	100.0	37.92	100.0

Screened Feed

Four tests were made in this series:

(1) The minus 14-plus 150-mesh screen fraction from the minus 8-mesh feed was pulped with water, floated with pine oil and cleaned 5 times (Test 24). After the second cleaning the minus 100-mesh material was wet screened from the concentrate before completing the test.

(2) The same procedure was followed in Test 25 except that the rougher concentrate was wet screened on a 65-mesh sieve before cleaning.

The results are given in Table 9.

(3) Minus 8-mesh feed was wet ground 5 minutes and screened on a 150-mesh sieve. The plus 150-mesh product was floated with pine oil and cleaned 5 times (Test 26, Table 10).

(4) The plus 14-mesh material was dry screened from minus 8 plus 150-mesh feed and passed through rolls. This size reduced fraction was combined with the minus 14-mesh, pulped with water, floated with pine oil and cleaned 5 times (Test 27). The last three cleaner tailings were fractionated by screening on 48-mesh sieves; plus fractions were combined (Table 11).

Air Tabling

Screened feed fractions were separated on a small Knapps and Bates air-table equipped with a sintered deck. Plus 14-mesh material, in which graphite was mostly attached, was not tried. Five finer fractions were tabled; minus 100-mesh feed was not suitable for treatment.

Results are shown in Table 12.

TABLE 9

Flotation Tests 24 and 25. Screened Feed, (-14+150 mesh)
Unground

Test	24	25
AGENT		
Pine oil	lb per ton	lb per ton
Rougher	0.1 x 3 = 0.3	0.1 x 3 = 0.3
Cleaner 1	0.1 x 2 = 0.2	0.1 x 2 = 0.2
Cleaner 2	0.1	0.1 x 2 = 0.2
Cleaner 3	0.1 x 2 = 0.2	0.1 x 2 = 0.2
Cleaner 4	0.1 x 2 = 0.2	0.1 x 2 = 0.2
Cleaner 5	0.1	0.1 x 2 = 0.2
REMARKS	Head dry screened on 14 mesh and wet screened on 150 mesh	
	Second cleaner concentrate wet screened on 100 mesh before further cleanings*.	Rougher concentrate wet screened on 65 mesh before cleaning**.

ANALYSES / Product	% Wt	% Carbon (LOI) Anal	% Carbon (LOI) Dist	% Acid Soluble Anal	% Acid Soluble Dist	% Wt	% Carbon (LOI) Anal	% Carbon (LOI) Dist	% Acid Soluble Anal	% Acid Soluble Dist
Plus 14 mesh	15.5	15.86	12.8	28.34	12.9	15.4	12.60	11.7	35.35	15.1
Minus 150 mesh	21.2	9.15	10.1	61.49	38.4	20.9	9.15	11.5	61.17	35.5
Concentrate	9.0	73.46	34.4	15.04	4.0	7.6	77.04	35.1	13.66	2.8
Cleaner 5 tailings	<0.1	35.64	<0.1	24.26	<0.1	0.1	***	0	24.41	0.1
Cleaner 4 tailings	0.1	← insufficient sample →				0.1	***	0	22.28	0.1
Cleaner 3 tailings	1.5	54.37	4.1	20.16	0.9	0.5	61.72	1.8	18.27	0.2
Minus 100 mesh*	1.8	57.43	5.5	24.19	1.3	-	-	-	-	-
Cleaner 2 tailings	1.3	62.03	4.2	17.44	0.7	1.5	79.58	7.4	11.44	0.5
Cleaner 1 tailings	1.3	31.56	2.1	30.12	1.1	2.8	37.95	6.4	24.59	1.9
Minus 65 mesh**	-	-	-	-	-	3.2	53.49	10.3	25.09	2.2
Rougher tailings	48.2	10.67	26.7	28.65	40.6	47.9	5.45	15.8	31.24	41.3
Head (calcd)	100.0	19.23	100.0	33.97	100.0	100.0	16.56	100.0	36.02	100.0

*** Insufficient sample

TABLE 10

Flotation Test 26. Screened Feed (-8+150 mesh)

Ground 5 minutes

REAGENT	lb per ton
Pine oil	
Rougher	0.1 x 3 = 0.3
Cleaner 1	0.1 x 2 = 0.2
Cleaner 2	0.1 x 2 = 0.2
Cleaner 3	0.1 x 2 = 0.2
Cleaner 4	0.1 x 2 = 0.2
Cleaner 5	0.1 x 2 = 0.2
REMARKS	Minus 8-mesh heads ground. Plus 150-mesh feed prepared by screening.

ANALYSES / Product	% Wt	% Carbon (LOI) Anal	% Carbon (LOI) Dist	% Acid Soluble Anal	% Acid Soluble Dist	Composition
Minus 150 mesh	29.3	8.09	13.7	61.44	47.9	
Concentrate	13.6	74.05	58.4	14.89	5.4	Free flake, middlings, some free gangue.
Cleaner 6 tailings	0.2	*	0	15.13	0.1	-
Cleaner 4 tailings	0.9	73.13	3.7	12.25	0.3	-
Cleaner 3 tailings	0.4	46.02	1.1	20.87	0.2	Mostly free gangue, some large free flakes.
Cleaner 2 tailings	1.3	71.39	5.6	14.13	0.5	-
Cleaner 1 tailings	1.0	17.79	1.0	30.01	0.8	-
Rougher tailings	53.3	5.33	16.5	31.56	44.8	Few middling particles, much free gangue.
Head (calcd)	100.0	17.23	100.0	37.53	100.0	

* Insufficient sample.

TABLE 11

Flotation Test 27, Screened Feed (-14+150 mesh)

Unground. Plus 14 mesh comminuted by rolls

REAGENT	lb per ton
Pine Oil	
Rougher	0.1 x 3 = 0.3
Cleaner 1	0.1 x 2 = 0.2
Cleaner 2	0.1 x 2 = 0.2
Cleaner 3	0.1 x 2 = 0.2
Cleaner 4	0.1 x 2 = 0.2
Cleaner 5	0.1 x 2 = 0.2
REMARKS	Tailings from cleaners 3, 4, and 5 screened on 48 mesh. Plus fractions combined*.

Product \ ANALYSES	% Wt	% Carbon (LOI) Anal	% Carbon (LOI) Dist	% Acid Soluble Anal	% Acid Soluble Dist
Minus 150 mesh	22.4	9.19	11.1	60.25	36.7
Concentrate	12.3	69.86	46.3	16.70	5.6
Plus 48 mesh, Cleaners 3,4 & 5 tailings*	1.1	80.11	4.6	11.38	0.3
Minus 48 mesh, Cleaner 5 tailings	0.1	7.87	<0.1	34.85	0.1
" " " Cleaner 4 tailings	0.2	8.49	0.1	33.37	0.2
" " " , Cleaner 3 tailings	0.2	**	0	35.18	0.2
Cleaner 2 tailings	1.7	74.72	6.9	13.25	0.6
Cleaner 1 tailings	2.6	39.94	5.7	26.06	1.9
Rougher tailings	59.4	7.84	25.2	33.69	54.4
Head (calcd)	100.0	18.50	100.0	36.76	100.0

* Large flakes, nearly all gangue present as middling particles.

**Insufficient sample.

TABLE 12

Analyses of Cut Fractions from Air Tabling Test 1

Fractions Tyler Mesh		% Wt	Cut No.	% Wt		% Carbon (LOI)		% Acid Soluble		Composition
Minus	Plus			Fraction	Overall	Anal	Dist	Anal	Dist	
	14	15.6	-	15.6	15.6	13.48	13.3	31.44	14.1	
14	28	16.8	1	0.5	0.1	76.02	0.5	6.41	0.1	Mostly free flake; few stained, some gangue (free).
			2	10.8	1.8	55.47	6.3	22.44	1.0	Same but more stained flake, middlings, free gangue.
			3	63.4	10.7	17.80	12.1	34.27	9.9	Mostly gangue, few free flakes, middlings.
			4	25.3	4.2	11.37	3.0	33.64	3.8	Gangue and middlings, few free flakes.
28	35	9.3	1	3.0	0.3	83.18	1.6	10.98	0.1	Mostly free flake, stained, middlings and free gangue.
			2	30.7	2.9	35.93	6.6	27.21	2.1	Mostly gangue, few free flakes, stained flake and middlings.
			3	53.4	4.9	14.34	4.5	29.14	3.8	Mostly gangue, combined particles, few free and stained flakes.
			4	12.9	1.2	5.17	0.4	25.83	0.8	Nearly all gangue or combined particle
35	48	10.3	1	5.7	0.6	85.71	3.3	9.82	0.2	Mostly free flake, few stained and middling flake, free gangue.
			2	43.6	4.5	26.40	7.5	29.64	3.6	Mostly free gangue, some free flake, some middlings.
			3	45.9	4.7	6.15	1.8	24.83	3.1	Mostly free gangue, few free and middling flakes.
			4	4.8	0.5	4.13	0.1	23.68	0.3	Gangue.

(Contd

Table 12 (contd)

48	65	8.9	1	4.3	0.4	73.41	1.9	15.91	0.2	Free flake, free gangue. Stained flake, few middlings.
			2	14.6	1.3	36.99	3.0	28.94	1.0	Free flake, more free gangue. Stained flake, few middlings.
			3	64.0	5.7	13.44	4.9	30.19	4.7	Very few free flake. Mica. Very few middlings. Mostly gangue.
			4	17.1	1.5	3.80	0.4	25.56	1.0	Almost all gangue.
65	100	9.0	1	3.7	0.3	85.80	1.6	12.22	0.1	Mostly free flake, some free gangue, few middlings.
			2	7.9	0.7	70.21	3.1	20.72	0.4	Free flake, free gangue. Middlings and stained flake.
			3	77.7	7.0	8.43	3.7	34.17	6.5	Mostly gangue, some mica, some free flake.
			4	10.7	1.0	2.69	0.2	25.74	0.7	Almost all gangue.
100		30.1	-	30.1	30.1	10.60	20.2	52.43	42.5	
Head (calcd)		100.0		-	100.0	15.78	100.0	37.10	100.0	

DISCUSSION

The method of comminution used to free flake graphite from the ore is important. Care must be taken to keep breakage minimal and not force gangue into soft graphite surfaces. Roll crushing, one of the accepted means of size reduction, can create the latter problem.

Self-grinding, the method tried with this soft, friable, oxidized ore, looks promising. Table 1 indicates that the addition of 1.1 pounds of plus 1-in. and 4.85 pounds of minus 1-in. plus 8 mesh material, (to original charges of 10 pounds of plus 1-in. and 8.5 pounds of minus 1-in. plus 8-mesh), produces 7.18 pounds of minus 8-mesh product per cycle of 18.5 minutes.

Examination of screen fractions of the self-ground material (Table 3) indicated most unbroken lumps were larger than 14 mesh in size.

When stage feeding was used to control frothing, pine oil floated the graphite cleanly. Some oil was lost in each cleaning; step additions were made as required. In practice, recirculation of middlings would lessen the amounts required.

In Test 13 (Tables 6 and 6A), when unground minus 8-mesh feed was floated with pine oil and the rougher concentrate treated ultrasonically before cleaning five times, the combined 5th cleaner tailings and non-magnetic fraction of the concentrate gave a recovery of 72.1% at a grade of 86.6% carbon. The calculated minus-28 plus 65-mesh size gave a recovery of 29.9% assaying 95.44% carbon.

In Test 15 (Table 8), when material ground for 15 minutes, was floated with pine oil, cleaned once, a recovery of 93.3% at a grade of 71.1% carbon resulted; the calculated non-magnetic fraction of the concentrate, in the minus 8- plus 65-mesh size range, gave an overall recovery of 46.1% of 93.47% carbon.

With pine oil, the flotation period is long - approximately 8 minutes in the roughers, and 4 minutes decreasing to 2 minutes in the cleaners. The use of other more surface-active collectors or frothers, with or without pine oil, probably would increase the rate of flotation of the flake; however, it would add to reagent costs, complicate a one-reagent circuit, and would produce a less clean float.

Pine oil is usually considered a frother or froth modifier with minimal collecting properties, but it is applicable here because graphite is easily made non-wettable. Clean, wettable gangue particles will not float, and middling particles can be dropped during cleaning.

Removal of the low-carbon magnetic fraction of the feed at the start of the circuit (Table 2) did not sufficiently benefit the non-magnetic products to be justified. With unground feed, magnetics removed from the floated concentrate upgraded the non-magnetic fraction (Test 12, Table 5 and Test 13, Table 6). With ground feed, magnetics removed from the concentrate of Test 15 (Table 8) showed improvement in grade of the finer sizes over Test 14 (Table 7).

The way the ore broke down by self-grinding (Table 3), where 12.2% of the carbon was in 23.1% of the minus 150-mesh fraction, suggests that discarding these fines by screening before flotation would be an economical procedure. The carbon lost would be too fine to have much value, slimes would be eliminated, and less pine oil would be used. The flotation circuit would be reduced in size, with a savings in capital cost.

The material appeared to separate well on an air table. In practice, larger cuts would be taken and cleaned once or twice to increase grade. Losses in finer sizes are high because free graphite blows away.

The sintered deck air table was suited to the type of closely sized separation tried. Air must be carefully controlled.

Free gangue could be largely eliminated by air tabling prior to using other methods of benefication. Close sizing probably would not be required for such an application.

The content of acid-soluble material in the concentrates could probably be reduced by acid treatment, but it may not be economically feasible.

What appeared as graphite under the microscope was often biotite, apparent when carbon was burnt off during analyses. Optical estimates of carbon content are therefore unreliable.

CONCLUSIONS

1. Self-grinding produced free graphite and biotite with minimum breakage of flakes and minor imbedded gangue minerals.

2. Air tabling of carefully screened fractions (minus 14 to plus 100 mesh) gave a good separation with concentrates grading 70 - 85% carbon. The combined products gave an analysis of 78.43% carbon with a recovery of 12%.

3. Flotation with pine oil, followed by screening, gave plus 65-mesh products containing:

95.4% carbon with 30% recovery, using unground feed.

93.5% carbon with 46% recovery, using ground feed.

Ultrasonics were used to free the graphite with the unground material. Removal of magnetics was used to upgrade the concentrates.

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REFERENCE

1. W.J.D. Stone, "Investigation of Concentration of Graphite from Mount Laurier, Quebec. Submitted by Dr. L. J. LaRue", IR 58-123 (July, 1958), Mines Branch, Department of Energy, Mines and Resources, Ottawa.

FHH/RAW/am

POOR

QUALITY

ORIGINAL